

# PATENT SPECIFICATION



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435,005

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## COMPLETE SPECIFICATION

### Process of Making Zinc Oxide

We, AMERICAN ZINC, LEAD & SMELTING COMPANY, a corporation of the State of Maine, United States of America, of 943, Paul Brown Building, St. Louis, Missouri, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to improvements in the manufacture of zinc oxide and more particularly to methods for controlling the shape of the particles. According to one well-known method for making zinc oxide a mixture of coal and zinc ore is spread upon a flat grate through a series of charging doors of a suitable furnace and at a high temperature (approximately 1400° C.). The zinc ore is reduced to metallic zinc and volatilized as a gas. As the zinc vapor, diluted with the products of combustion, passes over the charges of coal and zinc ore, it becomes oxidized through the action of air admitted to the furnace and, as it alternately meets fresh amounts of zinc vapor and air, the particles of zinc oxide that are formed grow into needle shape, as is clearly disclosed under the microscope. The relatively coarse pigment particles of zinc oxide follow an irregular path through the flue and are finally delivered by suction to a trail where they are cooled so that they can be safely filtered in a bag room.

In the operation of such a plant the charges of coal and zinc ore are delivered at periodic intervals to the grate, so that by the time the last charge is inserted through the last door in the series, the ash or clinker is ready to be withdrawn from the point where the first charge was inserted and a new charge is introduced. Under these conditions the characteristics of the product formed are continually changing; and further, weather conditions and other factors beyond the control of the furnace operation make it impossible to obtain a uniform product of the desired quality.

It has previously been proposed in specification No. 379,855, for the produc-

tion of zinc white from metallic raw materials, to vaporize the zinc in a furnace by means of hot gases or vapours which contain no constituents which react with or oxidize the zinc and which act directly on the molten zinc, the zinc vapour being then carried into an oxidizing region, the air-gas mixture thereafter being passed through condensation apparatus for separation of the zinc white.

According to the present invention, the furnace is operated under more uniform conditions. The charges of coal and zinc ore are introduced into the furnace as heretofore, but the draft of air to support combustion is limited and so regulated that the products, instead of being a mixture of zinc vapor and zinc oxide with CO, CO<sub>2</sub> and nitrogen, consist of no zinc oxide and a ratio of carbon monoxide to carbon dioxide sufficient to maintain the zinc in the elemental state at the furnace temperature, which is maintained at approximately 1200° C. This regulation of the draft of air results in an atmosphere practically completely non-oxidizing to zinc vapor above the charge in the furnace. The gases and zinc vapor under a slight positive pressure are then withdrawn from the furnace. In order to maintain the flow of gas uniformly, it is desirable to control the suction in the trail. The temperature and composition of the gases can be held very uniform and this plays an important part in the uniformity of the zinc oxide made by this process.

The gases from the furnace are passed to a suitable insulated tower or chamber where they are momentarily (one or more seconds) held at a high temperature, and thence to a point where air is either sucked or blown in under regulated volume and velocity, such that the zinc burns with a lazy flame. The amount of air introduced at this point is of importance and should be so controlled that the combined effect of the introduction of the air and the combustion of the zinc vapor will not reduce the temperature, though there may be a slight drop due to radiation losses and there may be an increase in temperature. The zinc oxide, as

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formed, and the zinc vapor slowly mix and form the desirable uniform long needles. The amounts and places of introduction of the auxiliary air are so controlled that after refining, (reheating) the properties of the oxide, such as for instance the oil absorption as determined by the Coleman-Gardner test, can be uniformly controlled as may be desired over the range of commercial requirements. The oil absorption property of the acicular zinc oxide thus produced can be varied by the size of the openings for the auxiliary air and the degree of laziness of the flame with which the zinc vapor burns.

For the purpose of enabling the invention to be better understood, reference is made to the accompanying drawing which is a diagrammatic view of one form of plant for making the acicular or needle shaped particles of zinc oxide.

In the accompanying drawing, in which like reference numerals indicate like parts throughout, 1 represents a furnace provided with any suitably perforated grate, either stationary or traveling, as may be desired, and 2 indicates charging openings for introducing coal and zinc ore into the furnace. The draft of air passing up through the grate for supporting combustion is regulated and controlled by any suitable means well known in the art, care being taken to so regulate this supply of draft air that the atmosphere above the grate shall be at all times a practically non-oxidizing atmosphere consisting of the products of combustion and the vaporized zinc. As here shown, the draft of air to the furnace is regulated or controlled by means of a fan 3 delivering air under low pressure to a conduit 4, and the passage of air from said conduit to the several sections of the furnace is controlled by suitable dampers 5. Due to blow holes in the charge there may be some small amount of oxygen get into the furnace, but this will be consumed by an excess of reducing gas in the furnace. Any zinc oxide particles formed under these conditions are small, and at the high temperature are easily reduced by the CO, so that the gases leaving the furnace consist of a mixture of CO, CO<sub>2</sub>, nitrogen and zinc vapor with practically no zinc oxide. The temperature and composition of these gases can be held very uniform and this plays an important part in the uniformity of the zinc oxide made by this process. The temperature in this furnace may vary somewhat above or somewhat below 1200° C. 6 indicates the usual or any suitable insulated tower into which the fumes from the furnace

pass through an opening 7 which is suitably controlled by baffles 8 to maintain a degree of back pressure in the furnace. The mixed products of combustion and zinc vapor, at a temperature of approximately 1200° C., pass from the tower 6 through a channel 9 and into a combustion chamber 10, from which chamber 10 they are drawn out, at a temperature of approximately 1100° C., through the trail 11 by means of a suction fan 12 and pass into the bag room 13. It will be apparent that, since the reaction involved generates heat when the concentrated zinc gases are oxidized, the results obtained would not be materially changed if the temperature in the tower 6 should be somewhat lower than that of the furnace, provided the temperature be held uniform. The rate of movement of the vapors from the furnace and of the zinc oxide particles and gases on the way to the bag room may be controlled by the speed of the fan 12, and if desired, by a suitable valve 14 introduced in the trail.

As the products of combustion and zinc vapor pass from the channel 9 into the chamber 10, air is introduced through openings 15 in the channel, and if desired, also through an opening 16 leading directly into the chamber 10. The air may be either sucked or blown in through the openings 15 and 16. Since it is introduced under very low velocity to the end that the zinc burns with a lazy flame, the zinc oxide particles as they form and the zinc vapor slowly mix with each other with the result that the acicular or needle shaped particles are built up. The size of the openings 15 and 16 for the admission of air and the degree of laziness of the flame in the chamber 10 where combustion of the zinc vapors occurs, are subject to control to the end that properties of the oxide, such as oil absorption, may be successfully controlled. The zinc oxide and gases of combustion enter the trail 11 from the combustion chamber 10 at a high temperature, approximately 1100° C., although the temperature may vary somewhat above or below this within a range that will not materially affect the results obtained. In addition to the gases that have been mentioned as produced in the furnace there may be small amounts of impurities, such as free sulphur, sulphur compounds, tars and finely divided carbon. While the gases from the furnace are in the insulated chamber 6 where they are maintained for a short time (one or more seconds) at a high temperature, the tars and carbon are removed, presumably by reaction with the carbon dioxide in the gas, and if

desired a small amount of air may be admitted through ports 17 at the entrance to chamber 6, care being taken not to admit sufficient air at this point to produce any appreciable amount of zinc oxide. This air however has the advantage of oxidizing sulphur and sulphur compounds into oxidized sulphur gases, and of more rapidly and completely removing tars and carbon.

It will be understood by those skilled in the art that the precise form of the plant indicated above may be varied without departing from the scope of the invention. Moreover, it will be recognized that the temperatures herein given may vary somewhat above or somewhat below those given, but it is to be particularly pointed out that in making the acicular or needle shaped particles of oxide, the temperature of the combined gases and oxide particles as they enter the trail have been but slightly lowered from that at which they leave the furnace.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The process of making zinc oxide of acicular form which consists in vaporizing zinc in a reducing atmosphere at approximately 1200° C. and then slowly mixing the zinc vapour with an oxidising agent outside the furnace while maintaining the temperature at a degree not materially above or below 1100° C.

2. The process as claimed in Claim 1 including refining the oxide.

3. The process of making zinc oxide of acicular form which consists in subjecting zinc ore to the action of burning coal in a closed furnace, so controlling the air draft to the furnace that the resulting zinc vapour is in a reducing atmosphere at a temperature of approximately 1200° C. in the furnace above the charge, passing the mixed zinc vapour and reducing gas to a zone outside the furnace in such amount that the temperature in said zone is maintained at a degree not materially above or below 1100° C., and introducing air at a very low velocity into the mixture, whereby the zinc vapour burns with a lazy flame and acicular zinc oxide particles are formed.

4. The process of making zinc oxide which consists in vaporizing zinc in a reducing atmosphere at a temperature of approximately 1200° C. in a furnace, withdrawing the zinc vapour and reducing gases from the furnace into an oxidizing zone while maintaining back pressure

in the furnace, and then slowly admitting air into the oxidizing zone in such amount that the temperature in said chamber is maintained at a degree not materially above or below 1100° C. and burning the zinc vapour with a lazy flame.

5. The process of making zinc oxide which consists in vaporizing zinc in a reducing atmosphere in a furnace at a temperature of approximately 1200° C., withdrawing the zinc vapour and reducing gases with any associated carbonaceous material into a less reducing atmosphere at approximately the same temperature, momentarily maintaining the temperature, whereby the impurities are eliminated, and then conducting said vapour and gases into a combustion zone and slowly admitting air into said zone in such an amount as to burn the zinc vapour with a lazy flame.

6. The process as claimed in Claim 5 which includes withdrawing the zinc vapours and reducing gases into a zone outside the furnace where they are momentarily maintained at approximately furnace temperature while air is slowly introduced in an amount such as not to oxidize the zinc vapours to any appreciable extent, whereby any associated carbonaceous materials are eliminated, and then passing the zinc vapours and reducing gases into the combustion zone, and slowly admitting air into said latter zone in an amount such that the temperature therein is not materially reduced thereby.

7. The process of making zinc oxide, which consists in vaporizing zinc in a reducing atmosphere, passing the zinc vapour into another zone into which a small amount of air is admitted, insufficient to oxidise any appreciable amount of the zinc vapours, and then passing the vapour and any oxide particles which have been formed in said zone into an oxidising zone wherein the air supply is regulated so as to ensure and control the growth of oxide particles.

8. The process as claimed in claim 7, which includes passing the zinc vapours through a combustion zone and slowly introducing an oxidising agent in said zone at different points along the length thereof.

9. The process as claimed in claim 5 or 6, including withdrawing the zinc oxide and gases from the combustion zone into a main or trail at a temperature of approximately 1100° C.

10. The process as claimed in claim 9, including maintaining the flow of zinc oxide and gases uniform by controlling the suction in the main or trail.

11. The improved process of making  
zinc oxide substantially as described.

Dated this 24th day of November, 1934.  
MARKS & CLERK.

Leamington Spa: Printed for His Majesty's Stationery Office, by the Courier Press.—1935.

[This Drawing is a reproduction of the Original on a reduced scale.]

